

Vertical Umbrella-Type Agitator to Promote Smooth Boiling in Vacuum Distillation

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AN UMBRELLA-TYPE agitator has been developed to promote smooth boiling in vacuum distillation.

It consists of a vertical glass rod with glass inverted cup-shaped baffles sealed to the rod at intervals along its length. Several holes are drilled in each baffle. The upper end of the vertical shaft consists of glass tubing, within which is a close-fitting soft iron core. To the bottom end of the shaft is attached a stainless steel bumper spring, which prevents damage to the agitator and the pot when the device is operating. Minor modifications permit location of the spring at the upper end of the agitator. When the external magnet is energized, the agitator moves upward, and when the circuit is broken it falls under the influence of gravity. A variable timer permits the number per of strokes unit time to be varied. A rate of 50 to 150 strokes per minute gave smooth steady boiling for all the liquids tested. The length of the stroke was such that at least one baffle broke the liquid surface regardless of the liquid level. A baffle spacing of 1 inch was satisfactory with 5-liter still pots. Heat was supplied by a standard Glascol mantle.

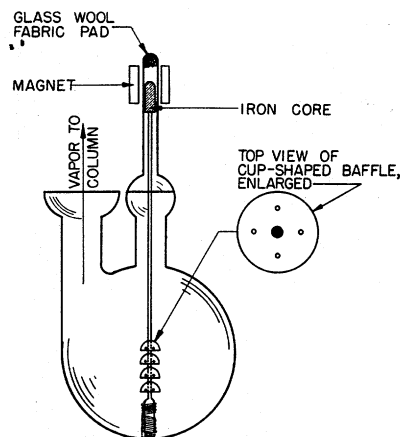
All the fatty acid esters and many of the acids and alcohols with an even number of carbon atoms from C_6 to C_{18} , inclusive, have been boiled successfully at high rates of vaporization and or long periods of time, at pressures that varied from 1.0 to 735 mm. of mercury. Water was present in some instances. The device has made possible the smooth steady boiling of methyl oleate and similar mixtures at a head pressure of 1 mm. of mercury for 170 hours.

Porcelain chips, broken glass, Boileezers, wooden chips, and an external hot spot could not be used where high rates of vaporization were required for lengthy periods. Internal hot spots (1) were not used because of concern as to possible decomposition. The use of a capillary bubbler was avoided because of the difficulty of obtaining completely dry and oxygen-free nitrogen or other inert gas and the uncertainty as to whether the presence of inert gas changed the phenomena of distillation under study.

The most satisfactory solenoids are those made for use with steam-duty electric valves (Magnatrol Valve Corp., 68 Fifth Ave., Hawthorne, N. J., Type 18A-42 solenoid coils for Magnatrol valve for steam service, with metal case wall). A coil of copper tubing may be made just to fit around the solenoid, and cooling water can then be circulated through the copper coil. The most satisfactory timer was the Model A1 Repeat-Cycle Timer with a terminal strip (C. G. Wilson Co., Chatham, N. J.).

The effectiveness of the agitator is believed to depend on continuous formation of new surface. On the upward stroke the baffle passes through the surface of the liquid and causes a new liquid surface to be formed. The downward stroke produces the same effect, and a small quantity of vapor is trapped beneath

the baffle and escapes through the holes in a stream of tiny bubbles. This again causes a new liquid surface to be formed. Hickman and coworkers (2) found that the rate of vaporization from a newly formed liquid surface is much greater than from an "old" surface. A rotary stirrer (3) also gives this effect to a limited degree.



In one test without the agitator, it was impossible to achieve smooth boiling over any appreciable period of time, with a 1-liter portion of commercial capric acid at 10 mm. of mercury pressure, regardless of the heat input. With the agitator, smooth steady boiling occurred for 14 hours, after which the test was stopped. The boil-up rate was approximately 2 or 3 liters of liquid per hour. A very marked decrease in rate was easily observed by watching the condensation ring in the condenser just after the agitator was momentarily stopped after smooth boiling had been obtained. The vapor stream practically ceased within 2 seconds after the agitator had been shut off and the liquid started to superheat. When the agitator was started again (before the first bump), rapid vaporization again commenced on the first stroke at an even faster rate than originally, until the superheat had been discharged.

LITERATURE CITED

- (1) Feldman, Julian, and Pantages, Peter, *ANAL. CHEM.*, **24**, 432 (1952).
- (2) Hickman, K. C. D., and Trevo, D. J., *Ind. Eng. Chem.*, **44**, 1882-911 (1952).
- (3) Lockwood, T. A., Le Tourneau, R. L., Matteson, Robert, and Sipos, Frank, *ANAL. CHEM.*, **23**, 1398 (1951).

A REPORT of work done under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act of 1946. The contract is being supervised by the Eastern Utilization Research Branch of the Agricultural Research Service. An application has been made for a patent covering the device described.